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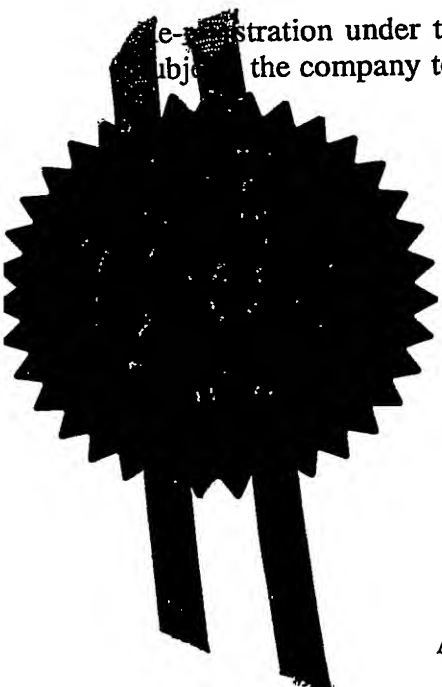
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1.	Your reference	SCB/RPS/58114/000

2.	Patent application number *(The Patent Office will fill in this part)*	**0201773.9**	28JAN02 E691007-18 D02882 _P01/7700 0.00-0201773.9

3.	Full name, address and postcode of the or of each applicant *(underline all surnames)*	Isis Innovation Limited
Ewert House, Ewert Place
Summertown
Oxford OX2 7SG
United Kingdom	25 JAN 2002
	Patents ADP number *(if you know it)*	3 99 856 4 003	
	If the applicant is a corporate body, give the country/state of its incorporation	United Kingdom	

4.	Title of the invention	X-ray Diffraction Method

5.	Name of your agent *(if you have one)*	**BOULT WADE TENNANT**
	"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*	**VERULAM GARDENS
70 GRAY'S INN ROAD
LONDON WC1X 8BT**
	Patents ADP number *(if you know it)*	42001

		Country	Priority application number *(if you know it)*	Date of filing *(day/month/year)*
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11   I/We request the grant of a patent on the basis of this application.

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[signature]   25 January 2002

12.   Name and daytime telephone number of person to   **Rohan Setna**
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X-ray Diffraction Method

The present invention relates to the field of X-ray diffraction of crystalline materials and, in particular, to an X-ray diffraction method for the determination of structural and/or chemical characteristics of polycrystalline engineering materials and components formed therefrom.

X-rays are electromagnetic radiation having a wave length of from 10^{-11} to 10^{-9} m and produced by bombardment of atoms by high-quantum-energy particles. In practice, X-rays may be produced by bombarding a metal target, for example copper or tungsten, with fast electrons in a vacuum tube.

The principle of X-ray generation in a vacuum tube is schematically illustrated in Figure 1. Electrons are emitted by the heated cathode (Ca) and accelerated by the applied voltage towards the anode (or anti-cathode, Ac) target. There they undergo rapid deceleration and absorption processes, which result in the emission of X-rays.

The radiation emitted as a result of the collision between electrons and the metal target can be separated into two components: (i) a continuous spectrum, which is spread over a wide range of wavelengths (also known as Bremsstrahlung); and (ii) a superimposed line spectrum, which is determined by the materials of the target (also known as the characteristic radiation).

The use of X-ray diffraction is of great importance in the analysis of crystals. For example, in the fields of metallurgy and materials science, X-

ray diffraction techniques may be used to identify the
lattice parameter and the structure of metal crystals.
Additionally, the techniques may be used to identify
the arrangement of different kinds of atoms in
5   crystals, the presence of imperfections, the
orientation of grains, the size of grains, the size
and density of precipitates and the state of lattice
distortion.

10   Known X-ray diffraction techniques include the
Laue method, the rotating crystal method and the
powder method (also known as the Debye/Scherrer
method).

15   In the Laue method a stationary single crystal is
bathed in a beam of polychromatic radiation. This
method is used for determining the orientation of
single crystals and the study of crystal
imperfections.
20

In the rotating crystal method a single crystal
is rotated in a beam of monochromatic X-rays. This
method is also used for the determination of crystal
structures.
25

In the powder method a powdered polycrystalline
sample is bathed in a beam of monochromatic radiation.
This method is used for the determination of lattice
parameters, grain sizes and the preferred orientation
30   of grains.

The powder method is also used to study bulk
polycrystalline samples, such as engineering
components and structures. For example, it can be
35   used to determine the lattice parameter variation in
orientation and position, and to deduce sample strain

and stress.

Conventional laboratory diffraction measurements rely on interrogating only a thin ($\approx$ < 50 μm) surface layer of the sample. Accordingly, such techniques do not provide information regarding stresses and strains in sub-surface regions or in the bulk of a sample.

Another widespread application of X-rays in industry and medicine is radiography, i.e. for transmission photography to obtain absorption contrast-images. The only property of the X-ray radiation that is utilised in such applications is the penetrating ability.

In standard radiographic set-up, industrial tungsten target X-ray tubes are used to produce polychromatic radiation within a widely divergent (~40°) cone.

In defectoscopic configuration, the object of study, for example an aluminium casting, is bathed in an incident X-ray beam, and a photographic plate or CCD camera is used to record the image which registers the attenuation of the beam along the path between the source and any given pixel. This set-up is only capable of providing information on relatively large defects (typically about a millimetre in size), such as casting voids.

For the purpose of quantitative diffraction analysis, the radiation produced by an industrial X-ray tube is not well suited in that it has a definition that is too poor both in terms of wave length (energy) and wave vector (direction).

The present invention aims to address at least some of the problems associated with the prior art.

In a first aspect the present invention provides an X-ray diffraction method for the analysis of polycrystalline materials, the method comprising:

(a) providing a polycrystalline material for analysis;

(b) providing a polychromatic X-ray source, wherein the source produces X-rays by accelerating charged particles to energies of no more than 1 MeV;

(c) collimating X-rays from the polychromatic X-ray source into a beam having a divergence in the range of from 10^{-4} to 10^{-2} radians;

(d) exposing at least a portion of the polycrystalline material to the collimated X-ray beam, whereby the beam is diffracted;

(e) collecting at least some of the diffracted X-rays; and

(f) analysing the collected, diffracted X-rays.

The following discussion applies to all aspects of the present invention unless otherwise stated.

The source preferably produces X-rays by accelerating charged particles to energies of no more than 500 keV, more preferably to no more than 400 keV, still more preferably to no more than 300 keV. The source will typically comprise means to accelerate electrons in a vacuum tube to impact a metal target.

The X-ray beam typically has a brilliance $\leq 10^{12}$ photons/s/mm^2/mrad^2/0.1% BW.

The diffracted X-rays may suitably be collected

by an energy dispersive X-ray detector or array. For example, a Li-drifted Si or Ge solid state detector may be used. The energy dispersive X-ray detector typically has a relative energy resolution of 0.5 x 10^{-2} to 4 x 10^{-2}, more typically 1 x 10^{-2} to 3 x 10^{-2}. An example of a suitable detector is the Canberra BEGe liquid nitrogen cooled, solid state energy dispersive germanium crystal X-ray and gamma-ray detector, in combination with multi-channel analyser, computer acquisition software and hardware.

The energy of the collimated X-ray beam is preferably ≥ 60 keV, more preferably in the range of from 100 to 300 keV. This enables the collimated X-ray beam to penetrate the polycrystalline material to a depth of typically ≥ 1 mm, preferably ≥ 5 mm, more preferably ≥ 10 mm, still more preferably ≥ 15 mm. Indeed, the attenuation depth can be up to 50 mm or more depending on the polycrystalline material. Thus, sub-surface X-ray analysis may be achieved. The attenuation depth is the thickness of material such that the transmitted beam intensity is equal to 37% of the incident beam intensity.

As mentioned above, the X-ray source will typically comprise means to accelerate electrons in a vacuum tube to impact a metal target. In this manner, a metal target is bombarded with fast electrons. The X-ray source is preferably an industrial X-ray source (for example a tungsten target X-ray tube) such as may be used in standard radiographic applications. A suitable example is a 160 kV, 1.6 kW industrial X-ray source (W tube) by Philips, supplied by AT Roffey Ltd (circa 1980).

The beam may be collimated using known

techniques.   For example, a pair of adjustable copper-
tungsten X-ray slits may be used.

The method may further comprise moving the
collimated X-ray beam relative to the polycrystalline
material.   In a preferred embodiment, the collimated
X-ray beam is scanned across at least a portion of the
polycrystalline material, while keeping the
polycrystalline material stationary.   This is
advantageous in circumstances where the
polycrystalline material forms part of a relatively
large engineering component.   In such cases, movement
of the component would be difficult.

The collected, diffracted X-rays may be analysed
in order to determine a structural and/or chemical
characteristic of the polycrystalline material, for
example lattice parameter determination.   In turn,
lattice parameter determination may be used to provide
information on stresses and/or strains in the
polycrystalline material and, preferably, to map
stresses and/or strains.   Stresses and/or strains in
the bulk of the polycrystalline material may be mapped
at a depth of typically $\geq$ 1 mm, preferably $\geq$ 5 mm,
more preferably $\geq$ 10 mm, still more preferably $\geq$ 15
mm.   Indeed, stresses and/or strains in the bulk of
the polycrystalline material may be mapped at a depth
of up to 50 mm or more depending on the
polycrystalline material.   This is in contrast to the
conventional laboratory techniques where only a thin
surface layer is analysed.

The polycrystalline material may be a natural
object or an engineering article or component part
thereof.   The polycrystalline material will typically
comprise a metal or alloy, a ceramic or a crystalline

polymer or a composite crystalline material, such as, for example, a ceramic reinforced metal matrix composite material. An example is SiC reinforced Al.

5   The polycrystalline material will typically have a thickness of $\geq$ 0.1 mm, more typically $\geq$ 1 mm, still more typically $\geq$ 5 mm, still more typically $\geq$ 10 mm, still more typically $\geq$ 15 mm. Indeed, the polycrystalline material may have a thickness of up to
10   50 mm or more depending on the absorption.

       In contrast to the conventional powder diffraction techniques, the polycrystalline material need not be in the form of a fine grained powder, nor is material removal, cutting or drilling of any kind
15   required within the region of interest. The method according to the present invention therefore allows engineering components to be analysed in their intended structural form. This is important for the
20   measurement of sub-surface engineering stresses and strains.

       In a second aspect, the present invention provides an apparatus for X-ray diffraction analysis
25   of polycrystalline materials, the apparatus comprising:
       (i)   a polychromatic X-ray source, wherein the source produces X-rays by accelerating charged particles to energies of no more than 1 MeV;
30   (ii)   means for collimating X-rays from the polychromatic X-ray source into a beam having a divergence in the range of from 10^{-4} to 10^{-2} radians;
       (iii)   an energy dispersive X-ray detector or array
35   for collecting at least some of the diffracted X-rays resulting, in use, from exposing at

least a portion of a polycrystalline material
to the collimated X-ray beam; and

(iv)   means for analysing the collected, diffracted
X-rays.

The polychromatic source may be moveable with
respect to a polycrystalline material to be analysed.
Advantageously, the collimated X-ray beam is adapted,
in use, to scan, across the sample of the
polycrystalline material, while the polycrystalline
material is maintained stationary.

In a third aspect, the present invention provides
a method of quantitatively mapping the sub-surface
distribution of the crystal lattice parameter in a
polycrystalline material, the method comprising:

(a)   providing a sample for analysis, wherein the
sample comprises a polycrystalline material;

(b)   providing a polychromatic X-ray source, wherein
the source produces X-rays by accelerating
charged particles to energies of no more than 1
MeV;

(c)   collimating X-rays from the polychromatic X-ray
source into a beam having a divergence in the
range of from 10^{-4} to 10^{-2} radians and an
attenuation length typically $\geq$ 1 mm;

(d)   scanning the collimated X-ray beam across the
sample, whereby the beam is diffracted;

(e)   collecting at least some of the diffracted X-rays
in an energy dispersive X-ray detector or array;
and

(f)   analysing the collected, diffracted X-rays to map
the lattice parameter in the polycrystalline
material.

The polycrystalline material may be a natural

material or, alternatively, an engineering material, including a component formed therefrom.

This method may preferably be used to determine the stresses and/or strains in polycrystalline natural or engineering materials and components formed therefrom. Accordingly, a preferred further step (f) involves transforming the map of the lattice parameter into a map of sub-surface engineering stresses and/or strains.

The technique of composite strain mapping is described in *Scripta Materialia*, Vol. 39, No. 12, pp. 1075-1712, 1988.

Typically, the present invention achieves a strain measurement accuracy of 50×10^{-6} to 200×10^{-6}, more typically 100×10^{-6} to 150×10^{-6}. The spatial resolution is typically from 0.25 mm to 1 mm.

The present invention will now be described further with reference to the following drawings, in which:

Figure 1 is a schematic illustration of the process of X-ray generation in a vacuum.tube;

Figure 2 shows a diffraction configuration used in the Example (from right to left – incident beam, slits, sample, slits, detector);

Figure 3 shows the X-ray source spectrum (energy) used in the Example;

Figure 4 shows the 'no sample' source pattern (lattice spacing) used in the Example;

Figure 5 shows a scattering pattern of a polycrystalline Al alloy sample used in the Example (log scale, counts);

Figure 6 shows a scattering pattern of a polycrystalline Al alloy sample used in the Example (linear scale, counts);

Figure 7 shows a Gaussian fit to the Al diffraction peak 1 (the box on the right gives the values and errors of the parameters); and

Figure 8 shows a Gaussian fit to the Al diffraction peak 2 (the box on the right gives the values and errors of the parameters).

Example

Transmission polychromatic X-ray diffraction experiments were carried out on 1.5 mm thick samples of rolled Al sheet. The strain accuracy achieved (prior to optimization) was approximately 150×10^{-6}. This corresponds to a stress accuracy of ≈ 10 MPa in aluminium, ≈ 14 MPa in copper, and ≈ 28 MPa in steel.

The experimental set-up comprised:

a)   A 160 kV, 1.6 kW industrial X-ray source (W tube) by Philips, supplied by AT Roffey Ltd (circa 1980), placed in a lead-lined room and position-controlled remotely using a traverse motor assembly;

b)   A Canberra BEGe liquid nitrogen cooled, solid state energy dispersive germanium crystal X-ray and gamma-ray detector, in combination with

multi-channel analyser, computer acquisition
software and hardware; and

c)   A pair of adjustable (0.5 to 5 mm) composite
5   (copper tungsten) X-ray slits.

For the purpose of spatial definition the beam
was collimated using two large blocks of lead, about
50 mm thick, which only allowed radiation through a
10   long slit, 0.5 mm wide. This set-up was used in the
diffraction experiment. However, a secondary slit was
needed to further define the beam direction, and also
as an anti-scatter device. The two slits were aligned
using a laser pointer. Similarly, a pair of slits was
15   used on the detector side to provide directional
definition. In both cases the slits were mounted at
two ends of a copper tube. Copper, a good photon
absorber, was used to reduce secondary scatter.

20   While the incident beam was close to horizontal,
the diffracted beam was defined to be inclined at an
angle of about 6.2°.

A schematic illustration of the set-up is shown
25   in Figure 2.

The lead block surfaces were irradiated by the
intense incident X-ray beam and thus fluoresced (i.e.
absorbed and re-emitted X-rays) at the characteristic
30   wavelengths (energies) of Pb. This is evidenced by
the energy spectrum shown in Figure 3. The
characteristic energies present due to the 'source'
properties do not change with the scattering angle
prescribed by the set-up. However, the peaks of
35   energy corresponding to diffraction from the crystal
lattice of the sample depend on the scattering angle

through Bragg's equation: $2d\sin\theta = n\lambda = hc/f$. This allowed the scattering angle to be adjusted so as to avoid, as far as possible, overlap with the 'signature' peaks of lead. Given a certain scattering

5   angle θ, the energy pattern is converted to the lattice parameter pattern using Bragg's equation. For the configuration used, $\theta=3.1°$, and the 'no sample' pattern is shown in Figure 4.

10   The sample was then placed at the intersection of the incident and scattered beams. The sample contributed to the flux into the detector, through diffraction on many individual grains. New peaks appear in the pattern. This is illustrated in Figure

15   5, where a logarithmic scale is used for the vertical axis to make the peak at approximately 2.3Å more prominent. Figure 6 shows the same profile using a linear vertical scale for the counts.

20   Although the aluminium diffraction peak at approximately 2.3Å in Figure 6 appeared broad and small, fitting it with a simple Gaussian function demonstrated that it provided acceptable accuracy. The Gaussian peak shape used for this peak (dubbed

25   'Peak 1') was described by the function:

m2*exp(-(m0-m1)^2/2/m3^2)+m4

Here m0 is the argument (lattice spacing) and m1

30   to m4 are the function parameters that are sought. The values of the parameters given above represent the initial guesses. The result of fitting is shown in Figure 7: Gaussian fit to the Al diffraction peak 1. The box on the right gives the values and errors of

35   the parameters.

The fitting result corresponds to a strain accuracy of $\Delta d/d=0.00033441/2.3304=145 \times 10^{-6}$. This corresponds to a stress accuracy of about 10 MPa in aluminium, 14.5 MPa in copper, and 29 MPa in steel.

The lattice spacing of 2.330 Å is close to the value of $4.05/\sqrt{3}=2.338$ Å for the (111) reflection in Al. The next reflection, (200), is expected at a lattice spacing of $4.05/2=2.025$ Å. Close inspection of Figure 6 does indeed show that a peak exists at approximately 2Å in that pattern, but is overlapped by a characteristic twin of lead. However, provided this twin is ignored in the analysis, this peak can be fitted as well, as shown in Figure 8: Gaussian fit to the Al diffraction peak 2. The box on the right gives the values and errors of the parameters.

The fitting result for peak 2 corresponds to a strain accuracy of $\Delta d/d=0.0013964/2.0105=700 \times 10^{-6}$. This corresponds to a stress accuracy of about 50 MPa in aluminium, 70 MPa in copper, and 140 MPa in steel. The accuracy is affected by the overlap with the characteristic twin of Pb. Information from multiple peaks may be combined to improve the overall accuracy.

The lattice spacing of 2.01 Å is close to the value of 2.025 Å for the (200) reflection in Al. The ratio of the spacing for the two peaks is 1.15, which is very close to the theoretically expected value of 1.156 for the face centred cubic (fcc) lattice.

This Example demonstrates that the accuracy of lattice parameter determination is adequate for engineering stress measurement applications. The rate of data collection was approximately 15 minutes per pattern.

In the present invention, polychromatic beam/energy dispersive detector X-ray transmission experiments are used to analyse polycrystalline engineering materials and components.

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The present invention provides a method of accurately determining the lattice parameter and thus stress measurements within the bulk of polycrystalline engineering materials and components.

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CLAIMS:

1.   An X-ray diffraction method for the analysis of polycrystalline materials, the method comprising:

(a)   providing a polycrystalline material for analysis;

(b)   providing a polychromatic X-ray source, wherein the source produces X-rays by accelerating charged particles to energies of no more than 1 MeV;

(c)   collimating X-rays from the polychromatic X-ray source into a beam having a divergence in the range of from 10^{-4} to 10^{-2} radians;

(d)   exposing at least a portion of the polycrystalline material to the collimated X-ray beam, whereby the beam is diffracted;

(e)   collecting at least some of the diffracted X-rays in an energy dispersive X-ray detector or array; and

(f)   analysing the collected, diffracted X-rays.

2.   A method as claimed in claim 1, wherein the source produces X-rays by accelerating charged particles to energies of no more than 500 keV.

3.   A method as claimed in claim 1 or claim 2, wherein the energy dispersive X-ray detector has a relative energy resolution of from 0.5×10^{-2} to 5×10^{-2}.

4.   A method as claimed in any one of the preceding claims, wherein the energy of the collimated X-ray beam is ≥ 60 keV, preferably in the range of from 100 to 300 keV.

5.   A method as claimed in any one of the preceding

claims, wherein the collimated X-ray beam penetrates the polycrystalline material to an attenuation depth of ≥ 1 mm.

5   6.   A method as claimed in any one of the preceding claims, further comprising moving the collimated X-ray beam relative to the polycrystalline material.

7.   A method as claimed in claim 6, comprising
10   scanning the collimated X-ray beam across at least a portion of the polycrystalline material, while keeping the polycrystalline material stationary.

8.   A method as claimed in any one of the preceding
15   claims, wherein the collected, diffracted X-rays are analysed in order to determine a structural and/or chemical characteristic of the polycrystalline material.

20   9.   A method as claimed in claim 8, wherein the structural characteristic is the lattice parameter.

10.   A method as claimed in claim 9, wherein lattice parameter determination is used to provide information
25   on phase distributions, stresses and/or strains in the polycrystalline material.

11.   A method as claimed in claim 10, wherein lattice parameter determination is used to map phase
30   distributions, stresses and/or strains in the polycrystalline material.

12.   A method as claimed in claim 11, wherein lattice parameter determination is used to map phase
35   distributions, stresses and/or strains in the polycrystalline material at a depth of ≥ 1 mm.

13.   A method as claimed in any one of the preceding claims, wherein the polycrystalline material is an engineering article or component part thereof.

14.   A method as claimed in any one of the preceding claims, wherein the polycrystalline material comprises a metal or alloy, ceramic or crystalline polymer, including combinations of two or more thereof.

15.   A method as claimed in any one of the preceding claims, wherein the polycrystalline material is a composite material comprising a crystalline phase.

16.   A method as claimed in claim 15, wherein the metal matrix composite material is a glass and/or ceramic reinforced metal matrix composite material.

17.   A method as claimed in any one of the preceding claims, wherein said portion of the polycrystalline material has a thickness of $\geq$ 1 mm.

18.   An apparatus for X-ray diffraction analysis of polycrystalline materials, the apparatus comprising:
(i)   a polychromatic X-ray source, wherein the source produces X-rays by accelerating charged particles to energies of no more than 1 MeV;
(ii)   means for collimating X-rays from the polychromatic X-ray source into a beam having a divergence in the range of from 10^{-4} to 10^{-2} radians;
(iii)   an energy dispersive X-ray detector or array for collecting at least some of the diffracted X-rays resulting, in use, from exposing at least a portion of a polycrystalline material to the collimated X-ray beam; and
(iv)   means for analysing the collected, diffracted

X-rays.

19.   An apparatus as claimed in claim 18, wherein the polychromatic source is moveable with respect to a polycrystalline material to be analysed.

20.   An apparatus as claimed in claim 18 or claim 19, wherein the collimated X-ray beam is adapted, in use, to scan, across the polycrystalline material, while the polycrystalline material is maintained stationary.

21.   A method of quantitatively mapping the sub-surface distribution of the crystal lattice parameter in a polycrystalline material, the method comprising:

(a)   providing a sample for analysis, wherein the sample comprises a polycrystalline material;

(b)   providing a polychromatic X-ray source, wherein the source produces X-rays by accelerating charged particles to energies of no more than 1 MeV;

(c)   collimating X-rays from the polychromatic X-ray source into a beam having a divergence in the range of from 10^{-4} to 10^{-2} radians, and a penetration depth of $\geq$ 1 mm;

(d)   scanning the collimated X-ray beam across the sample, whereby the beam is diffracted;

(e)   collecting at least some of the diffracted X-rays in an energy dispersive X-ray detector or array; and

(f)   analysing the collected, diffracted X-rays to map the lattice parameter in the polycrystalline material.

22.   A method as claimed in claim 21, wherein the polycrystalline material is a natural material or an engineering material, including a component formed

therefrom.

23.   A method as claimed in claim 21 or claim 22, further including:

5   (f)   transforming the map of the lattice parameter into a map of sub-surface engineering stresses and/or strains.
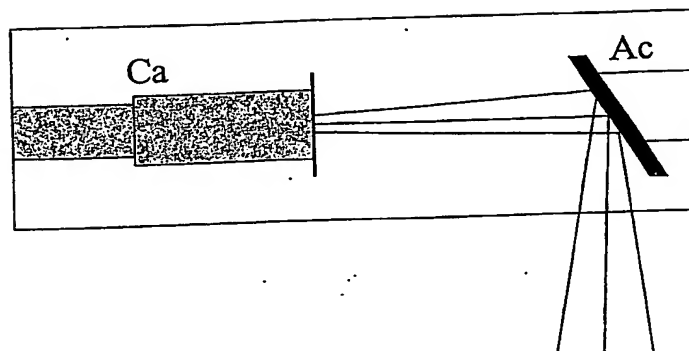
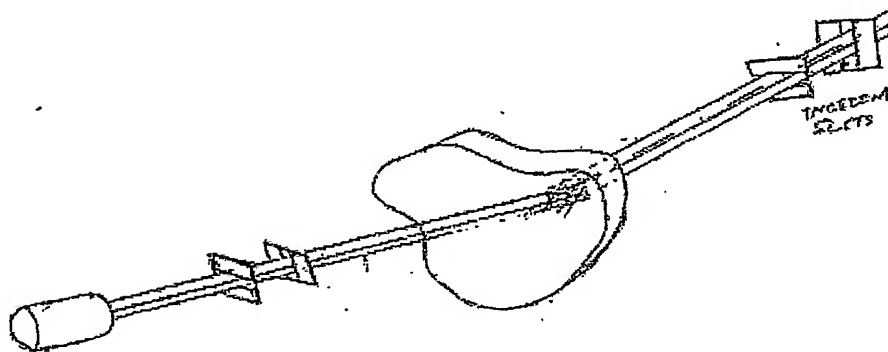Figure 1. Schematic illustration of the process of X-ray generation in a vacuum tube.

Figure 2. Diffraction configuration (right to left): incident beam – slits – sample – slits – detector.
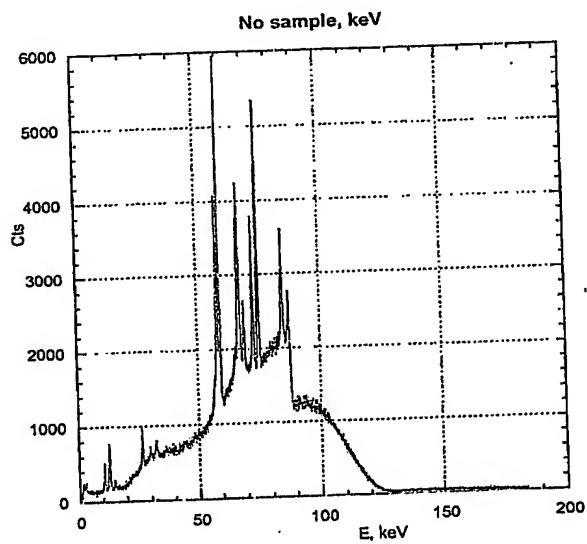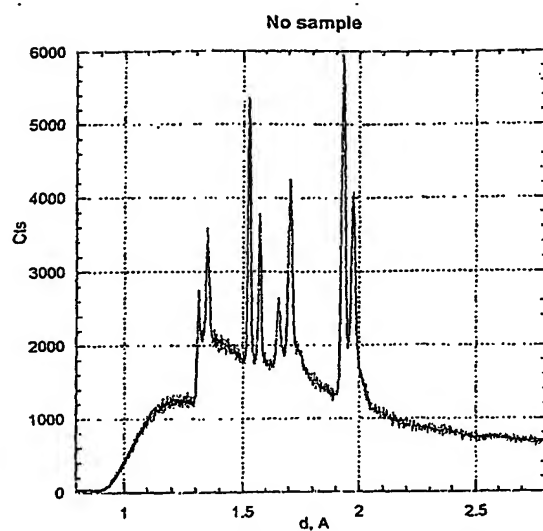

Figure 3. The 'source' spectrum (energy).

No sample

Figure 4. The 'source' pattern (lattice spacing)

Whole pattern (Log counts)
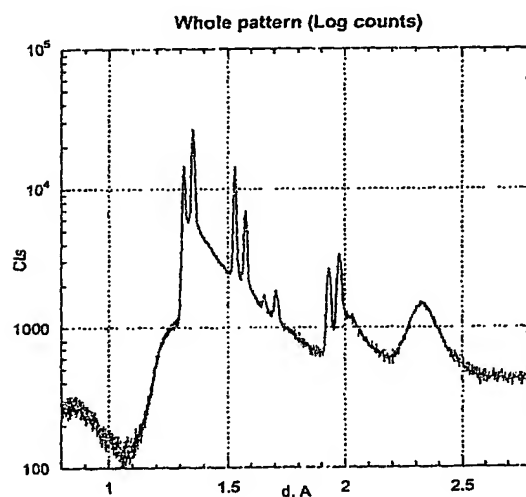
Figure 5. Scattering pattern of a polycrystalline Al alloy sample (log scale, counts).

Whole pattern (Lin counts)
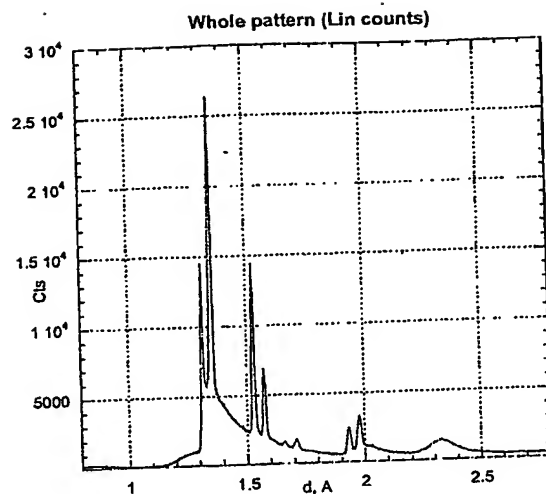
Figure 6. Scattering pattern of a polycrystalline Al alloy sample (linear scale, counts).
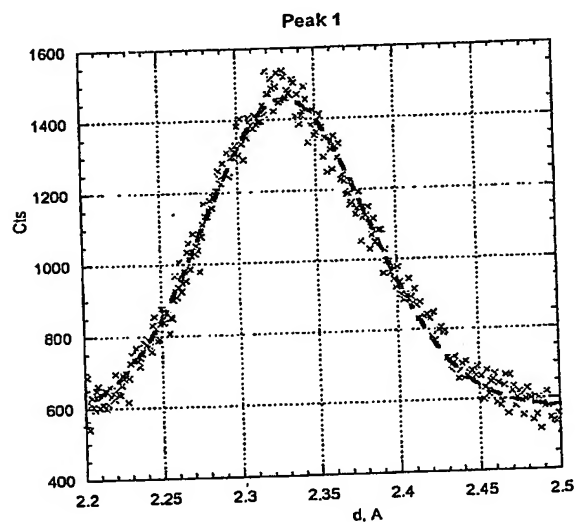
Peak 1

$y = m2^*exp(-(m0-m1)^2/2/m3^{\wedge}...$		
	Value	Error
m1	2.3304	0.00033441
m2	886.11	6.7817
m3	0.052465	0.00057504
m4	572.99	6.1367
Chisq	3.8277e+05	NA
R	0.99281	NA

Figure 7.
Gaussian fit to the Al diffraction peak 1. The box on the right gives the values and errors of the parameters.

y = m2*exp(-(m0-m1)^2/2/m3^...		
	Value	Error
m1	2.0105	0.0013964
m2	546.57	9.2459
m3	0.046939	0.0015414
m4	650.02	7.4931
Chisq	2.0525e+05	NA
R	0.98723	NA

Figure 8.